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IS 5044 (1969): Benzanthrone [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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Indian Standard
SPECIFICATION FOR BENZANTHRONE

(First Reprint AUGUST 1991)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *June* 1969

Indian Standard
SPECIFICATION FOR BENZANTHRONE

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(*Continued from page* 1)

Drafting Panel for Anthraquinone, CDC 46 : P7

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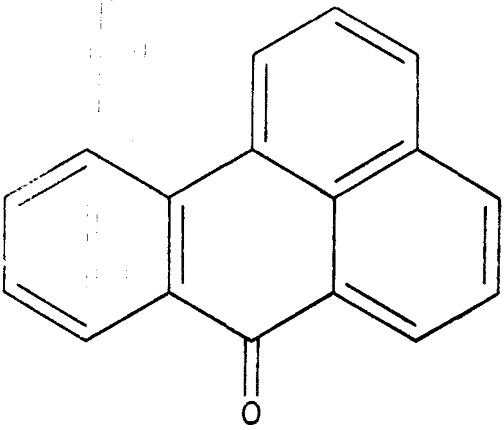
Indian Standard
SPECIFICATION FOR BENZANTHRONE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 13 February 1969, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 Benzanthrone is an important dye intermediate mainly used for vat and disperse dyes. It has the following structural formula.



(Benzanthrone, Molecular Weight 230·2)

0.3 This standard is one of a series of Indian Standards on dye intermediates. A complete list of such standards is given on P 14.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for benzanthrone.

---

*Rules for rounding off numerical values (*revised*).

2. GRADES

2.1 There shall be two grades of benzanthrone, namely, pure and technical.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of yellow (for pure) or olive green (for technical) powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR BENZANTHRONE

Sl No.	Characteristic	Requirements		Methods of Test (Ref to Cl No. in Appendix A)
		Pure	Technical	
(1)	(2)	(3)	(4)	(5)
i)	Moisture content, percent by weight, *Max*	0·2	0·5	A-1
ii)	Ash, percent by weight, *Max*	0·2	10	A-2
iii)	Anthraquinone content, percent by weight, *Max*	0·5	0·5	A-3
iv)	Benzanthrone content, percent by weight, *Min*	98·0	80	A-4

4. PACKING AND MARKING

4.1 Packing — The material shall be suitably packed in polyethylene bags or as agreed to between the purchaser and the supplier.

4.2 Marking — The containers shall be securely closed and marked with the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Batch number; and
d) Tare, gross and net weights.

4.2.1 Each container shall, in addition, bear the minimum cautionary notice worded as under:

AVOID CONTACT WITH SKIN; MAY CAUSE ALLERGIC DERMATITIS

4.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in Appendix B.

6. TEST METHODS

6.1 The tests shall be carried out according to the methods prescribed in Appendix A, as indicated in col 4 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1, and *Clause* 6.1)

METHODS OF TEST FOR BENZANTHRONE

A-1. DETERMINATION OF MOISTURE CONTENT

A-1.1 Apparatus

A-1.1.1 *Weighing Bottle* — squat type, conforming to IS : 1574-1960†.

A-1.1.2 *Desiccator* — containing an efficient desiccant, such as phosphorus pentoxide.

A-1.1.3 *Air-Oven* — preferably electrically heated, with temperature control device.

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*Specification for water, distilled quality (*revised*).

†Specification for glass weighing bottles.

A-1.2 Procedure — Weigh accurately about 10 g of the material into the weighing bottle which has been previously dried and weighed. Place the weighing bottle in the air-oven for approximately one hour at 105° ± 1°C. Remove the bottle from the oven, cool in the desiccator to room temperature and weigh. Repeat this procedure of heating, cooling and weighing until the difference between two successive weighings does not exceed 1 mg.

A-1.3 Calculation

$$\text{Moisture content, percent by weight} = \frac{100\,w}{W}$$

where

w = loss in weight in g of the material upon drying, and
W = weight in g of the material taken for the test.

A-2. DETERMINATION OF ASH

A-2.1 Apparatus

A-2.1.1 *Silica Crucible*

A-2.1.2 *Desiccator*

A-2.2 Procedure — Heat the silica crucible in a muffle furnace, cool in a desiccator and weigh. Repeat the operations of heating, cooling and weighing until constant weight is obtained. Weigh accurately 2 g of the sample into the crucible and gently ignite until most of the material has burnt out. Place the crucible in a muffle furnace at about 800°C until no more carbon particles are visible. Cool in a desiccator and weigh. Repeat the operations of heating, cooling and weighing till constant weight is obtained.

A-2.3 Calculation

$$\text{Ash, percent by weight} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

where

W_3 = weight of the crucible along with the ash,
W_1 = weight of the silica crucible, and
W_2 = weight of the crucible along with the sample.

A-3. DETERMINATION OF ANTHRAQUINONE CONTENT

A-3.0 Outline of the Method — The material is dissolved in toluene and reduced with sodium hydrosulphite. The depth of the resulting red colouration due to leuco-anthraquinone is compared with prepared colour standards.

A-3.1 Reagents

A-3.1.1 *Sodium Hydroxide Solution* — 1 percent (*w/v*).

A-3.1.2 *Sodium Hydrosulphite*

A-3.1.3 *Toluene* — *See* IS : 537-1967*.

A-3.2 Procedure — Dissolve 0·2 g of sample in 60 ml toluene by warming in a 100-ml conical flask. Transfer the solution to a 250-ml separating funnel. Add 100 ml of sodium hydroxide solution heated to 80° to 85°C. Add 1 g of sodium hydrosulphite and shake vigorously. Leuco-anthraquinone shows red colouration. Allow separation into layers. Collect the lower aqueous layer in a 100-ml conical flask. Heat to boiling to remove turbidity. Compare the red colour developed with special colour standards prepared as described in **A-3.3** and preserved in sealed 100-ml test-tubes.

A-3.3 Preparation of Special Colour Standards — Prepare colour standard for the anthraquinone test as follows:

a) *Colour Standard ' A ' for 0·0 Percent Anthraquinone* — Dissolve 0·3 g of potassium chromate (K_2CrO_4) in 500 ml of water.

b) *Colour Standard ' B ' for 0·5 Percent Anthraquinone*

  1) Dissolve 0·2 g of potassium dichromate ($K_2Cr_2O_7$) in 500 ml of water.

  2) Dissolve 25·0 g of cobalt nitrate [$Co(NO_3)_2.6H_2O$] in 250 ml of water.

  Mix 25 ml of solution 1, 8 ml of solution 2 and 5 ml of water.

c) *Colour Standard ' C ' for 1·0 Percent Anthraquinone*

  1) Dissolve 1·0 g of potassium dichromate ($K_2Cr_2O_7$) in 500 ml of water.

  2) As in **A-3.3** (b) (2).

  Mix 13·5 ml of solution 1 and 16·5 ml of solution 2.

A-4. DETERMINATION OF BENZANTHRONE CONTENT

A-4.0 Outline of the Method — The purity of the sample is estimated by employing the chromatographic method. Benzanthrone is separated chromatographically and determined quantitatively by spectrophotometer.

A-4.1 Reagents

A-4.1.1 *Alumina* — chromatographic grade. To get a material of desired activity, treat the alumina as follows.

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*Specification for toluene, pure, nitration grade (*revised*).

A-4.1.1.1 Prepare a solution containing 20 to 30 mg of pure benzanthrone in 100 ml of ethylene dichloride. Set up a chromatographic tube as described in **A-4.2.1** and chromatograph the solution. A compact main band shall be obtained; it shall be well separated from any earlier or later bands; and it shall be completely washed through the column with the addition of not less than 50 ml and not more than 150 ml of the eluent. If the chromatogram is unsatisfactory in any of these three respects and the alumina is insufficiently active, reject it. If the alumina is too active, deactivate it as follows:

> Spread out the alumina evenly on a stainless steel tray in a thin layer. Add the necessary amount of water slowly from a burette covering the whole of the tray as evenly as possible. Mix the alumina by hand initially and then transfer to a Kilner jar and mix by rolling mechanically for at least two hours. Then retest for activity. If still too active repeat the procedure adding more water in increments of 0·5 percent.

A-4.1.2 *Ethylene Dichloride* — Dry for 24 hours over anhydrous calcium chloride, filter and distil. Collect the portion boiling between 83° and 84°C. The optical density fraction shall not exceed 0·005 at 385 mμ (*see* IS : 869-1956*).

NOTE — Ethylene dichloride is toxic. Proper precautions should be taken to avoid inhalation of vapours and contact of the liquid with skin.

A-4.1.3 *Benzanthrone* — chromatographically pure.

A-4.1.4 *Preparation* — Dissolve 0·1 g of technically purified benzanthrone in minimum amount of ethylene dichloride. Transfer this solution gradually to the chromatographic tubes prepared with alumina and ethylene dichloride and described in **A-4.2.1.** Allow the solution to pass through the column and elute further by ethylene dichloride. Reject coloured bands of impurities and collect the lemon yellow band of benzanthrone. Keep a battery of 5 or 6 tubes in operation for collecting a sizable quantity of pure material.

Concentrate the solution of pure benzanthrone by evaporation until crystallization occurs on cooling to room temperature. Filter off the crystals and dry to constant weight in a vacuum oven at 100° ± 5°C.

To check the purity of the material, dissolve about 0·01 g (accurately weighed) in minimum amount of ethylene dichloride and pass the solution through a chromatographic column set up as described in **A-4.2.1**. Elute as necessary with solvent. Collect the band carefully and determine the optical density at 385 mμ. Make up the solution of purified crystals using the same amount of crystals and solvent. Determine its optical density in the same way. A difference in optical densities of more than 0·003 indicates that impurity is present in the chromatographically purified benzanthrone. In that case repeat the chromatographic purification until this check test is satisfied.

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*Specification for ethylene dichloride, technical.

A-4.2 Apparatus

A-4.2.1 *Chromatographic Column* — Set up vertically a glass tube 40 cm long and about 1·5 cm internal diameter, joined with a 50-ml thistle funnel at the upper end and fitted with a stop-cock at the lower end. Place a cotton-wool plug in the tube and press to the bottom of the tube by means of a glass rod flattened at the end. Place a disc of filter paper cut to the approximate internal diameter of chromatographic tube on top of the cotton-wool.

A-4.2.1.1 Prepare a slurry of about 15 g of alumina in ethylene dichloride and pour it into the tube. Wash down the side of the tube and pack the column by light tapping. Place first a disc of filter paper and then a cotton-wool plug at top surface of alumina.

Always keep enough solvent in the column so that it remains wet and that at least 2 cm of solvent layer always persists over the top of alumina. On no account allow the alumina to run dry. In event of this happening, reslurry the alumina and repack.

A-4.2.2 *Spectrophotometer*

A-4.2.3 *Standard Calibration Graph* — Prepare a number of standard solutions of chromatographically pure benzanthrone in ethylene dichloride varying in concentration from 0·5 mg/100 ml to 2·5 mg/100 ml with a difference of 0·2 mg/100 ml between any two successive concentrations.

Take readings of optical density or percentage transmittance of the above mentioned solutions at wavelength of 385 mμ using the special optically matched cells specified for the particular spectrophotometer used. The temperature of the solutions immediately before and after measurement shall be 27° ± 2°C.

Where the spectrophotometer is calibrated only in percentage transmittance the optical density may be obtained by reference to standard conversion tables. Plot the calibration graph of concentration (in terms of mg/100 ml of solvent) versus optical densities. The curve obtained becomes the standard calibration graph.

NOTE 1 — The readings of optical density or percentage transmittance are taken at the wavelength of maximum absorption. This is obtained by plotting absorption against wavelength, and determining the peak of the curve thus obtained.

NOTE 2 — The spectrophotometer shall be checked for accuracy from time to time.

A-4.3 Procedure

A-4.3.1 *For Pure Grade* — Weigh 120 mg to 150 mg of sample accurately in a 100-ml clean dry conical flask. Add about 50 ml of ethylene dichloride and reflux for 10 to 15 minutes. Cool and transfer the solution to a 100-ml volumetric flask. Wash the conical flask well with ethylene dichloride and transfer the washings to the flask. Dilute the solution to 100-ml mark. Drain the excess solvent from the column so that the top remains just wet. Transfer with the help of a burette 10 ml of the sample solution accurately and slowly to the top of a chromatographic tube (*see* **A-4.2.1**) dropwise and at the same time drain the solvent from the column at the same rate without ever allowing the top of the column to become dry. Fill the upper part of the tube with solvent and allow the chromatogram to develop, keeping a good head of solvent above the alumina throughout. Reject the coloured bands preceding the main (lemon-yellow coloured) band of benzanthrone. Start collecting fractions when the main band is about 1·5 cm from the lower tip of the tube. When the entire benzanthrone band is eluted, dilute to 250 ml exactly. From this solution take accurately 25 ml solution and dilute to 100 ml exactly in a volumetric flask. This is the test solution.

Set the wavelength of the spectrophotometer to the predetermined value (*see* Note 1 under **A-4.2.3**), and adjust the instrument in such a manner that the transmittance through the blank becomes 100 percent after inserting the cell with blank solution. Replace the cell with the solution of the sample and read the optical density or percentage transmittance. In case of latter refer to the standard conversion tables and find out the corresponding optical density. Read the concentration C against this optical density from the standard calibration graph (*see* **A-4.2.3**).

A-4.3.2 *For Technical Grade* — Weigh accurately 200 mg to 250 mg of sample in a 100 ml clean dry conical flask. Pour about 50 ml of ethylene dichloride and reflux for 10 to 15 minutes. Cool and transfer the solution to a 100-ml volumetric flask, keeping the solid behind, as much as possible. Carefully wash this solid several times with small portions of ethylene dichloride till the washing is colourless. Add washings to the main solution and dilute this to 100-ml mark.

Follow hereafter the same procedure as given for benzanthrone, pure grade in **A-4.3.1**.

A-4.4 Calculation — The purity of benzanthrone (for both pure and technical grades) is calculated as follows:

$$\text{Benzanthrone, percent by weight} = \frac{C \times 10^4}{W}$$

where

C = benzanthrone content in mg per 100 ml of the test solution read from the calibration graph, and

W = the weight of the sample in mg taken for test.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF BENZANTHRONE

B-1. GENERAL REQUIREMENTS

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed, as thoroughly as possible, by suitable means.

B-1.5 The sample shall be placed in a clean, dry and air-tight glass or other suitable containers on which the material has no action. The containers shall be stored in a cool and dry place.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars that may be necessary for identification of the consignment.

B-1.8 Benzanthrone can cause allergic dermatitis. Proper precaution shall be taken during sampling to avoid contact with skin.

B-2. SAMPLING INSTRUMENT

B-2.1 The sampling instrument is a closed type sampling tube, undivided, consisting of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel) closely fitting into each other throughout their entire length so that one tube can be rotated within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference are cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sampler is usually provided with locking arrangements so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate the entire depth of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm proportionately to the length. A length of 150 cm and a diameter of 30 mm will satisfy most needs.

B-2.1.1 *Use of Sampling Instrument* — The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes and finally closing them withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further cores shall be taken from different parts of the same container such that they are at least 75 mm in the case of drums, bags, etc, and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

B-3. SCALE OF SAMPLING

B-3.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, then the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

B-3.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected for drawing the samples shall depend upon the size (N) of the lot and shall be in accordance with Table 2.

B-3.3 These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used. In case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them in one order as 1, 2, 3……..., etc, up to r and so on, where r is the integral part of N/n (N being the lot size and n the number of containers to be selected). Every rth container thus counted shall be withdrawn to give sample for test.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clause* B-3.2)

LOT SIZE N (1)	NO. OF CONTAINERS TO BE SELECTED n (2)
4 to 25	3
26 ,, 50	4
51 ,, 100	5
101 ,, 150	6
151 ,, 300	7
301 and above	8

NOTE — When the size of the lot is three or less, the number of containers to be selected and the criteria for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

B-4. TEST SAMPLE AND REFEREE SAMPLE

B-4.1 From each of the containers selected according to **B-3**, draw with an appropriate sampling implement small portions of the material from different parts of the container.

B-4.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

B-4.3 From the samples obtained according to **B-4.2** representing different containers, equal quantities of material shall be taken and mixed thoroughly to form a composite sample, sufficient to carry out triplicate testing for all the characteristics. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, one for the supplier and the third for the referee.

B-4.4 All the composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **B-1.7**.

B-4.5 The referee composite sample, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-5. NUMBER OF TESTS

B-5.1 Tests for the determination of all the characteristics specified in this standard shall be conducted on the composite sample.

B-6. CRITERIA FOR CONFORMITY

B-6.1 For declaring the conformity of the lot to this standard, the test results for each of the characteristics shall satisfy the relevant requirement specified in this standard.

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*Sales Office in Calcutta is at 5 Chowringhee Approach, P. O. Princep Street, Calcutta 700072 — 27 68 00

†Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007 — 89 65 28

‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square, Bangalore 560002 — 22 36 71

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 APRIL 1972
TO
IS : 5044-1969 SPECIFICATION FOR BENZANTHRONE

Addenda

(*Page* 4, *Table* 1)

a) *Sl No.* (iv) — Add the following after Sl No. (iv) under the respective columns:

(1)	(2)	(3)	(4)	(5)
v)	Melting point, °C	166-169	—	—

b) *Col* 5 — Add the following new column after column (5) and also insert the foot-note with (*) mark:

SL No.	METHOD OF TEST (REF TO CL NO.)
(1)	(6)
i)	—
ii)	—
iii)	—
iv)	—
v)	4 of IS : 5299-1969*

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*Methods of sampling and tests for dye intermediates.

(CDC 46)